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# Electron Capture Gas Chromatographic Determination of Kepone Residues in Environmental Samples

Health Effects Research Lab, Research Triangle Park, N C

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## Electron Capture Gas Chromatographic Determination of Kepone® Residues in Environmental Samples

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**Abstract.** The pollution of the environment with Kepone® (decachlorooctahydro-1,3,4-metheno-2*H*-cyclobuta[*cd*]pentalen-2-one) by an industrial manufacturer of the pesticide resulted in the contamination of several terrestrial media, including biological life, near the entry of the insecticide into the ecosystem.

The substrata investigated and for which residue methodology was developed included river sediment, soil, water, shellfish, and finfish. Rigorous extraction techniques utilizing the Soxhlet apparatus and the Polytron® tissue homogenizer were required for complete removal of Kepone from the samples. Finfish tissue was the most difficult to analyze. For this type of substratum, a preliminary cleanup by gel permeation chromatography was required to remove most of the lipid material followed by a micro Florisil® column elution to eliminate polychlorinated biphenyls (PCBs). Cleanup of shellfish and other environmental samples was accomplished with a micro Florisil column only. Electron capture gas chromatography was used to analyze the sample extracts. Recoveries of Kepone from fortified samples averaged 84% or greater.

### Introduction

The pesticide Kepone®<sup>1</sup> (decachlorooctahydro-1,3,4-metheno-2*H*-cyclobuta[*cd*]pentalen-2-one) was developed in the early 1950's by the Allied Chemical Company. Most of the Kepone produced has been exported to certain foreign countries for use on bananas and potatoes. The compound has a number of registered uses in the United States, but is predominantly used in ant and roach baits. It has recently come under intense scrutiny from a methodology and toxicology viewpoint because of the discovery, in 1975, of environmental contamination by this compound near its place of manufacture in Virginia, U.S.A.

This study represents an investigation into the development of suitable analytical methodology using Kepone-containing field samples. Determinations

<sup>1</sup> This pesticide is also known as chlordane. Since most readers are probably more familiar with the trade name Kepone, it will be used throughout this article.

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for the compound were made in several different environmental media obtained from the proximity of a pesticide plant.

Few articles on analytical methodology for the pesticide appear in the literature. Other articles pertain to the evaluation of insecticidal properties of Kepone derivatives (Gilbert *et al.* 1966a, Gilbert *et al.* 1966b) and elucidation of the structure of Kepone or similar compounds (McBee *et al.* 1956, Griffen and Price 1964, Eaton *et al.* 1960). Gilbert *et al.* (1966b) prepared numerous alcohol and amine adducts of Kepone in a search for compounds possessing insecticidal activity. Determination of residues on vegetation of a similarly structured pesticide, known as GC-9160 (Kelevan), was accomplished by oxidation to Kepone and subsequent gas chromatography with electron capture (EC-GC) and electrolytic conductivity detection (Westlake *et al.* 1970). Brewerton and Slade (1964) presented a procedure for the analysis of Kepone residues on apples, utilizing EC-GC. They reported 0.2 ng as being readily detected and measured; however, most of the Kepone levels reported in their article were in the ppm range. Determination of Kepone in milk from cows fed treated feed was reported to be quantitative at 0.005 ppm (Smith and Arant 1967). Cleanup of samples with oleum and dilute base was followed by GC with microcoulometric detection.

The choice of solvents is critical in the extraction, cleanup and analysis for Kepone. Also, unless great care is taken to exclude water from solvents, the compound exists in solution as the hydrate. Aliphatic hydrocarbon solvents such as hexane are poor solvents for this pesticide; methanol, acetone, and benzene are better solvents. Acetone has been reported to react with Kepone under 18 hr of reflux (Gilbert *et al.* 1966a). At ambient temperature methanol apparently reacts with Kepone to form the hemiketal. Combined gas chromatography - mass spectrometry (GC/MS) indicates that the hydrate and hemiketal forms revert to Kepone in the injection port of a gas chromatograph at temperatures above 200°C.

Due to the polar nature of Kepone, it was difficult to remove it from substrata using nonpolar extraction solvents such as petroleum ether or hexane. During our investigation of several environmental media we found that the most effective extraction solvent systems were methanol-benzene (1:1) and toluene-ethyl acetate (1:3).

Three gas chromatographic columns were used: 3% OV-1, 1.5% OV-17/1.95% QF-1, and 4% SE-30/6% OV-210. Usually the 3% OV-1 column exhibited fewer gas chromatographic interferences in addition to giving a satisfactory retention time for Kepone. Retention times relative to aldrin ranged from 2.5 to 2.8, depending upon the column and the operating temperature.

## Experimental

### Apparatus<sup>2</sup>

Tracor, MT-220 or MT-222, gas chromatographs equipped with Nickel-63 electron capture detectors were operated in the d.c. or pulsed linearized mode. Borosilicate glass columns (1.8 m. × 4 mm i.d.)

<sup>2</sup> Use of trade names is for identification purposes only and does not constitute endorsement by the U.S. Environmental Protection Agency.

were packed with 80/100 mesh Gas Chrom Q which were coated with 1.5% OV-17/1.95% QF-1, 3% OV-1, or 4% SE-30/6% OV-210. Columns were operated at 200 to 210°C with a nitrogen or 5% methane in argon flow rate of 60 to 80 ml/min. Detector, inlet, and transfer line temperatures were 250, 235, and 220°C, respectively.

An N-Evap (Organomation Assoc., Worcester, MA) was used for reducing solvent volumes.

Willem's Polytron® Type PT 20 (distributed by Brinkman Instruments Inc., Westbury, NY).

Gel Permeation chromatograph, Autoprep Model 1001 (Analytical Biochemistry Laboratories, Inc.) equipped with a 250 or 350 × 25 mm glass column containing 200 to 400 mesh Bio-Beads (S-X3).

Vortex-Genie mixer, 115 volts, Scientific Industries, Inc.

Hobart food chopper, Model 84142, Hobart Manufacturing Co., Troy, OH.

Centrifuge, Model HN-S, International Equipment Co., Needham Heights, MA.

Blendor, Laboratory, Waring Products Co., Winsted, CT.

The following glassware was used: 40-ml round bottom centrifuge tubes, 25-ml graduated tubes, Kontes size 2525 (K570050); Chromaflex columns, 200 × 7 mm id, reservoir volume 50 ml, size 22 (Kontes 420530); Soxhlet Extractor size 50 × 250 mm, 15-ml graduated centrifuge tubes; disposable Pasteur pipets, 23-cm length, Kuderna-Danish (KD) concentrator assembly (K-5700000) fitted with a 25-ml graduated tube; Snyder column.

#### Reagents and Materials

Kepone® analytical reference standard, 87.3%.

Sodium Sulfate, anhydrous, granular, Soxhlet extracted for 6 hr with benzene and oven-dried at 130°C.

Floril® (60-100 mesh) was used in a form activated by the manufacturer at 1200°F.

Benzene, methanol, hexane, toluene, ethyl acetate, and acetonitrile were pesticide quality or equivalent.

#### Extraction

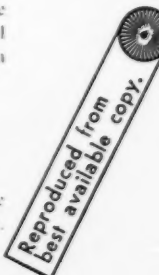
**Sediment and Soil.** A 20-g aliquot of air dried (24 to 48 hr) sample was extracted in a Soxhlet apparatus for 16 to 18 hr using 300 ml of methanol-benzene (1:1). The extract was concentrated and adjusted to volume in a 100-ml volumetric flask. If preliminary GC analysis indicated a Kepone level below 0.5 µg/g, a Floril column cleanup was usually necessary prior to quantitative determination by EC-GC.

#### Finfish and Shellfish

A homogeneous sample of eviscerated fish was prepared with a Hobart food chopper. Approximately six clams or oysters were thawed, shucked and drained prior to mixing in a food blender for sufficient time to obtain a homogeneous sample. The tissue was stored at -10°C until analyzed. A 10 g sample of the homogenate was weighed into a 40-ml round bottom tube and extracted with a Polytron tissue homogenizer using 25 ml of toluene-ethyl acetate (1:3) for approximately 2 min. After extraction the sample was centrifuged at 1500 rpm and the supernatant transferred through anhydrous Na<sub>2</sub>SO<sub>4</sub> into a 100-ml volumetric flask. The extraction was repeated twice more, combining the extracts; the volume was adjusted to 100 ml. An aliquot of fish extract equivalent to 1.0 g of tissue was cleaned up with gel permeation chromatography to remove the lipids. Subsequent elution through a micro Floril column was necessary to remove polychlorinated biphenyls (PCBs).

#### Water

Samples were collected and stored under refrigeration in one-liter jars fitted with Teflon® liners. The container was shaken vigorously just prior to measuring a 50-ml aliquot with a graduated cylinder.



The sample was poured into a 125 ml separatory funnel, 5 ml of benzene was added and the funnel shaken for 2 min. After phase separation, the aqueous layer was drained into the graduated cylinder and the benzene layer percolated through anhydrous  $\text{Na}_2\text{SO}_4$  into a 15-ml graduated centrifuge tube. The water was returned to the separatory funnel and the graduated cylinder rinsed with the two 2.5 ml portions of benzene, adding each to the separatory funnel. The sample was shaken again, the phases allowed to separate, and the benzene combined with the first extract. The volume was adjusted so that Kepone would be within linear range of the detector. Special care was taken to insure that the extract to be injected into the GC contained at least 1% methanol. Florisil column cleanup was not always necessary but depended on the sample.

#### *Sample Cleanup*

**Preparation and Elution of Florisil Column.** A chromaflex column (size 22-7) was loosely plugged with a small amount of glass wool. Florisil (1.6 g) was added in small increments with tapping. Anhydrous, granular  $\text{Na}_2\text{SO}_4$  (1.6 g) was added on top of the Florisil. Thirty ml of methanol-benzene (1:1) was used to wash the column free of interferences. After thoroughly air drying, the column was placed in an oven at 130°C overnight prior to use.

A prepared column was removed from the oven and allowed to cool before beginning sample elution. The column was wetted with several ml of solvent I (hexane containing 2% methanol and 4% benzene). When the solvent level reached the top of the  $\text{Na}_2\text{SO}_4$ , an aliquot of sample not exceeding 500 mg in 0.2 to 0.3 ml was placed on top of the column. Quantitative transfer of sample was accomplished with three 0.5-ml rinsings with solvent I. A first fraction of 7 ml of the above solvent was collected and discarded. PCBs and most of the common organochlorine pesticides were eluted in this fraction. The Kepone was collected by eluting with 30 ml of solvent II (hexane containing 1% methanol, 2% acetonitrile, and 4% benzene). The eluate was adjusted to an appropriate volume for GC injection. Methanol was added to the sample after evaporation of solvent in an amount necessary to bring the concentration to 1%.

#### *Gel Permeation Chromatography*

The sample (1 to 2 g) contained in 10 ml of toluene-ethyl acetate (1:3) was introduced into the GPC Model 1001 Autoprep. The sample was eluted with toluene-ethyl acetate (1:3) at a rate of approximately 5 ml/min. The first 106 ml was channeled to waste and the next 68 ml (containing the Kepone) was collected in a KD assembly with a 25-ml graduated tube. A Snyder column was attached and the solvent was concentrated to approximately 20 ml on a steam bath. Further evaporation of the solvent, almost to dryness, was accomplished with an N-Evap at 65°C. The sample was then transferred to a Florisil column and eluted as previously described.

### **Results and Discussion**

Methanol enhanced and stabilized the electron capture gas chromatographic response of Kepone dissolved in nonpolar solvents. The response reached a maximum at approximately 1% methanol content and remained constant; an increased percentage of methanol did not elevate the response. This was apparently due to the formation of the corresponding hemiketal which is less likely to adhere to surfaces in a microliter syringe. It was found that Kepone could not be removed from a syringe used to inject solutions made with hexane or benzene containing no methanol. Therefore, hexane and benzene solutions of standards and samples were adjusted to at least a 1% methanol concentration prior to injection into the gas chromatograph. Solvents which allowed maximum

response without the presence of methanol were: acetone, ethyl acetate, and toluene-ethyl acetate (1:3).

The quantitative removal of Kepone from several different types of environmental samples was accomplished using rigorous extraction with polar solvents. Due to the highly polar nature of the solvents, extraneous substances were also extracted, and made cleanup more difficult. Successive extractions of Kepone from samples was utilized to ascertain optimum extraction conditions. Values in Table 1 show complete extraction of the pesticide from water samples after two partitionings with benzene. Recovery data in Table 2 for distilled water and river water samples reveals that the methodology yields quantitative results for fortified samples. Data in Table 1 indicate that benzene alone did not completely extract Kepone from sediment in 3 hr with a Soxhlet extractor, since a significant amount was subsequently recovered by extracting an additional 3 hr with methanol-benzene (1:1). A third 3 hr extraction with methanol-benzene (1:1) on the same sample yielded only a trace of Kepone. These data also show that a

Table 1. Efficiency of Kepone extraction

| Sample                   | Solvent                     | Extraction time, hr | Kepone found $\mu\text{g/g}^a$ | Extraction method |
|--------------------------|-----------------------------|---------------------|--------------------------------|-------------------|
| Water                    | benzene                     |                     | 2.54 ng/g                      | Partitioning      |
|                          | benzene                     |                     | < 0.04 ng/g                    | Partitioning      |
| Sediment <sup>b</sup>    | benzene                     | 3                   | 1.00                           | Soxhlet           |
|                          | methanol-benzene (1:1)      | 3                   | 0.82 <sup>c</sup>              | Soxhlet           |
|                          | methanol-benzene (1:1)      | 3                   | trace                          | Soxhlet           |
| Sediment <sup>b</sup>    | benzene                     | 3                   | 0.22                           | Soxhlet           |
|                          | methanol-benzene (1:1)      | 3                   | 0.06                           | Soxhlet           |
| Soil <sup>b</sup>        | methanol-benzene (1:1)      | 6                   | 13.000                         | Soxhlet           |
|                          | methanol-benzene (1:1)      | 3                   | 3.05                           | Soxhlet           |
| Soil <sup>b</sup>        | methanol-benzene (1:1)      | 18                  | 9.34                           | Soxhlet           |
|                          | methanol-benzene (1:1)      | 18 <sup>e</sup>     | 0.05                           | Soxhlet           |
| Fish (shad)              | toluene-ethyl acetate (1:3) |                     | 1.51                           | Polytron          |
|                          |                             |                     | 0.01                           | Polytron          |
|                          |                             |                     | (4th extr.)                    |                   |
| Fish (bass) <sup>d</sup> |                             |                     | < 0.01                         | Polytron          |
|                          |                             |                     | (5th extr.)                    |                   |
|                          | acetone-acetonitrile (1:5)  |                     | 2.22                           | Polytron          |
| Oyster                   | toluene-ethyl acetate (1:3) |                     | 2.63                           | Polytron          |
|                          | toluene-ethyl acetate (1:3) |                     | 0.31                           | Polytron          |
|                          | toluene-ethyl acetate (1:3) |                     | < 0.01                         | Polytron          |
|                          |                             |                     | (4th extr.)                    |                   |

<sup>a</sup> Data for water expressed as ng/g

<sup>b</sup> The same sample aliquot was successively extracted with the solvent(s) indicated

<sup>c</sup> This aliquot was acidified with dilute HCl and re-extracted

<sup>d</sup> Different aliquots of the same tissue homogenate were extracted

single extraction of soil for at least 6 hr with methanol-benzene (1:1) was quantitative for Kepone.

Since Kepone is more soluble in alkaline than acidic media, a previously extracted soil sample (pH 8.1) which contained 9.3 ppm Kepone, was re-extracted after acidification (below pH 5). Less than 1% additional Kepone was removed after acidification. However, most of the sediment and soil samples were below pH 8. A Florisil column was quite effective in removing many of the extraneous substances in soil and sediment samples which produced a response with the electron capture detector. Sludge samples behaved similarly to soil and sediment in extraction and cleanup characteristics. Figure 1 compares gas chromatograms of a representative sediment sample before and after Florisil column cleanup.

The recovery data for Kepone fortified soil and sediment samples in Table 2 indicate complete recovery of Kepone from sediment after 18 hr Soxhlet extraction with methanol-benzene (1:1). It was satisfactory and convenient to arrange for overnight extraction of the samples.

The extraction efficiency of Kepone from fish tissue was investigated by making successive extractions with the Polytron<sup>®</sup> tissue homogenizer. This device was used by Johnson and Starr (1972) for extraction of pesticides from soil. Eviscerated shad was extracted the usual three times with the Polytron

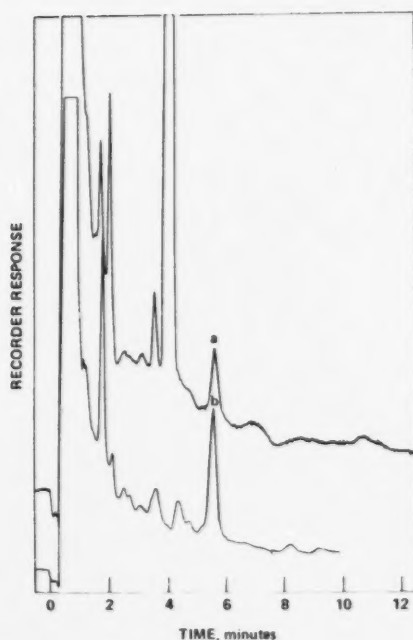


Fig. 1. Chromatograms of (a) sediment sample extract without cleanup. Injection: 600  $\mu$ g sediment extract equivalent. (b) Sediment sample extract with Florisil cleanup. Injection: 3 mg sediment extract equivalent. Column: 1.5% OV-17/1.95% QF-1 on 80/100 mesh Gas Chrom Q. Oven temperature 200°C.  $N_2$  flow rate 80 ml/min

Table 2. Recovery of Kepone from fortified samples

| Sample                | Fortification level, $\mu\text{g/g}^a$ | Extraction solvent          | Extraction method | Recovery, % |
|-----------------------|----------------------------------------|-----------------------------|-------------------|-------------|
| Water (distilled)     | 2 ng/g                                 | benzene                     | Partitioning      | 90          |
| Water (distilled)     | 2 ng/g                                 | benzene                     | Partitioning      | 92          |
| Water (distilled)     | 2 ng/g                                 | benzene                     | Partitioning      | 96          |
| Water (river)         | 5 ng/g                                 | benzene                     | Partitioning      | 90          |
| Water (river)         | 5 ng/g                                 | benzene                     | Partitioning      | 92          |
| Soil <sup>b</sup>     | 1.0                                    | methanol-benzene (1:1)      | Soxhlet           | 86          |
| Sediment <sup>c</sup> | 0.1                                    | methanol-benzene (1:1)      | Soxhlet           | 99          |
| Oyster                | 0.3                                    | toluene-ethyl acetate (1:3) | Polytron          | 84          |
| Fish (bream)          | 0.1                                    | toluene-ethyl acetate (1:3) | Polytron          | 94          |
| Fish (bream)          | 0.1                                    | toluene-ethyl acetate (1:3) | Polytron          | 80          |
| Fish (bream)          | 0.1                                    | toluene-ethyl acetate (1:3) | Polytron          | 92          |

<sup>a</sup> Fortification of water expressed as ng/g<sup>b</sup> 3-hr extraction (no Florisil column cleanup)<sup>c</sup> 18-hr extraction (no Florisil column cleanup)

Note: Kepone was not affected by boiling for 18 hr in methanol-benzene 1:1

using toluene-ethyl acetate (1:3) and quantitative recovery of Kepone was attained; fourth and fifth extractions of the same sample yielded less than 1% additional pesticide. A comparison of extraction solvents indicated a superiority of toluene-ethyl acetate over acetone-acetonitrile (1:5), as significantly more Kepone was obtained in an extraction with the former solvent (Table 1) from a aliquot of the same fish tissue sample homogenate. Toluene-ethyl acetate was initially utilized because it could be introduced directly into the gel permeation chromatograph (GPC).

Recovery experiments were run on water, soil, sediment, shellfish, and fish samples by fortifying with Kepone standard. Soxhlet extraction of soil for 3 hr was not sufficient for complete Kepone recovery. Removal of Kepone from fortified fish and shellfish using Polytron extraction was fairly rapid (*ca.* 1 hr/sample) and procedural recovery was quantitative.

Since total determination of Kepone depended upon quantitative recovery through both extraction and cleanup procedures, it was necessary to establish the recovery characteristics of Kepone through a Florisil column. The values in Table 3 show the results of elution of Kepone standards through a Florisil column using solvent II. At very low levels (5 to 30 ng) the recovery was only semi-quantitative but at higher amounts it was at least 90%. An initial 7-ml elution with solvent I was necessary in fish determinations to remove interference from PCBs in the gas chromatograph analysis; no PCB interference was observed in the other environmental samples.

Several aliquots of two (Kepone free) sediment extracts were fortified at various levels and eluted through a Florisil column to ascertain the recovery and degree of cleanup obtained. The results (Table 4) indicate quantitative recovery even though less solvent than usual was collected. However, in general, at least

Table 3. Typical recoveries of Kepone standards from Florisil columns

| Kepone added, ng | Elution solvent         | ml | Kepone found, ng | Recovery, % |
|------------------|-------------------------|----|------------------|-------------|
| 5.0              | Solvent I <sup>a</sup>  | 7  | 3.4              | 68.0        |
|                  | Solvent II <sup>b</sup> | 30 |                  |             |
| 30.0             | Solvent I               | 7  | 25.3             | 84.3        |
|                  | Solvent II              | 30 |                  |             |
| 60.0             | Solvent I               | 7  | 57.9             | 96.5        |
|                  | Solvent II              | 30 |                  |             |
| 100.0            | Solvent I               | 7  | 90.7             | 90.7        |
|                  | Solvent II              | 30 |                  |             |

<sup>a</sup> Solvent I - Hexane containing 2% methanol and 4% benzene<sup>b</sup> Solvent II - Hexane containing 1% methanol, 2% acetonitrile, and 4% benzene

30 ml of Solvent II was used for quantitative elution of Kepone through Florisil for the other samples investigated.

A Florisil column cleanup of oyster tissue extract was usually adequate for EC-GC analysis. As much as 500 mg of oyster tissue extract was eluted through the column. Figure 2 illustrates the gas chromatograms of an oyster tissue extract prior to and after Florisil column cleanup. GPC (Johnson *et al.* 1976) was used as a cleanup technique for fish tissue extracts. A Florisil column was not by itself satisfactory because of the inordinate amount of lipid material co-eluted with Kepone. GPC removed a high percentage of the fish lipids, but the PCBs which were present eluted with Kepone constituting an interference in the gas chromatographic analysis. The PCBs were removed with a Florisil column. Figure 3 depicts the cleanup achieved on a fortified fish sample carried through GPC and a Florisil column. This particular sample was one of the most difficult to analyze due to the high level of PCBs and lipid material. Recovery of standards through GPC averaged about 95%; however, the lower results recorded for actual fish samples was due to the necessity of additional elution through a Florisil column to remove PCB interferences (Table 5).

Table 4. Recovery of Kepone through Florisil columns from fortified sediment sample extracts<sup>a</sup>

| Sample | Sample size, g | Fortification level |      | Solvent, ml |    | Recovery, % |
|--------|----------------|---------------------|------|-------------|----|-------------|
|        |                | µg                  | µg/g | I           | II |             |
| 1      | 0.6            | 2                   | 3.33 | 5           | 25 | 109         |
|        | 0.6            | 0.4                 | 0.67 | 5           | 25 | 89          |
|        | 0.6            | 0.04                | 0.07 | 5           | 25 | 89          |
| 2      | 1.2            | 4                   | 3.33 | 5           | 25 | 96          |
|        | 1.2            | 0.8                 | 0.67 | 5           | 25 | 90          |
|        | 1.2            | 0.08                | 0.07 | 5           | 25 | 97          |

<sup>a</sup> Aliquots of Kepone-free sediment sample extracts were used

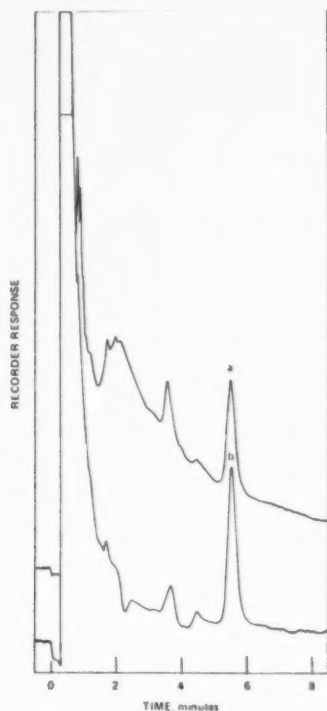


Fig. 2. Chromatograms of (a) Kepone from oysters extract without cleanup. Injection: 100  $\mu$ g tissue extract equivalent. (b) Oysters extract with Florisil cleanup. Injection: 200  $\mu$ g tissue extract equivalent. Column: 1.5% OV-17 1.95% QF-1 on 80/100 mesh Gas Chrom Q. Oven temperature 200°C.  $N_2$  flow rate 80 ml/min.

Kepone was confirmed in selected environmental samples by combined GC/MS. Chemical ionization using methane reagent gas produced a fairly strong  $M+1$  molecular ion cluster in the region  $m/e$  487 to  $m/e$  497. Simultaneous monitoring of four ions in the cluster and the characteristic GC retention time of Kepone added substantial confidence to the identification.

### Conclusions

Detailed methodology for determination of Kepone residues in several different types of environmental samples has been presented. Extraction by Soxhlet apparatus and the Polytron extractor was found to be entirely adequate for removal of Kepone in the substrata investigated. The Polytron extraction of fish and shellfish samples was quicker than other commonly employed extraction techniques and gave excellent results. A micro Florisil column cleanup prior to EC-GC analysis was adequate for all media investigated except fish tissue extracts. Gel permeation chromatography was utilized for these samples to

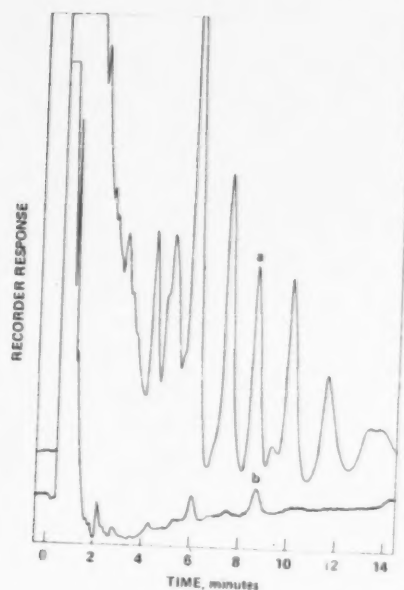


Fig. 3. Chromatograms of (a) Kepone from fortified fish tissue extract (0.1 ppm) without cleanup. Injection: 200  $\mu$ g of tissue extract equivalent. (b) Kepone from fortified fish tissue extract (0.1 ppm) after GPC and Florisil column cleanup. Injection: 200  $\mu$ g tissue extract equivalent. Column: 3% OV-17 on 80/100 mesh Gas Chrom Q. Oven temperature 200°C.  $N_2$  flow rate 80 ml/min.

Table 5. Recovery of Kepone from fortified fish tissue extracts after gel permeation chromatography and Florisil column cleanup<sup>a</sup>

| Kepone added, ng | Kepone concentration, $\mu$ g/g | Recovery, % <sup>b</sup> |
|------------------|---------------------------------|--------------------------|
| 100              | 0.10                            | 85.5                     |
| 100              | 0.10                            | 85.5                     |
| 100              | 0.10                            | 82.2                     |
| 100              | 0.10                            | 83.6                     |
| 100              | 0.10                            | 85.2                     |
| 100              | 0.10                            | 84.3                     |
| 100              | 0.10                            | 82.7                     |
| 100              | 0.10                            | 80.0                     |

<sup>a</sup> An amount of sample equivalent to 1 g of extracted fish tissue was used for each determination

<sup>b</sup> Range 80.0 to 85.5%. Average 83.6%. Standard Deviation  $\pm 1.9\%$

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remove most of the lipids and was followed by a micro Florisil column cleanup to remove PCBs.

The procedures presented in this study are a significant and valuable contribution to Kepone residue methodology because of the wide range of environmental media analyzed. Using the methodology presented, the minimum practical level of detection for Kepone was generally 40 ppt in water, 10 to 20 ppb in sediment, soil, fish and shellfish samples.

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